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20. ABSTRACT (Continue on reverse side if necessary and identify by block number) The report represents an initial effort to detoxify war gas identification sets with agent adsorbed on charcoal. This test involved a small pilot operation in which 31 bottles of agent on charcoal were added to 9.4 gallons of MEA in a 15-gallon drum. The procedure involved rolling the drum twice a day for 1 hour. This lasted for 8 days, at which time the necessary DOT tests were run. The work performed indicates that the Navy sets and replacement sets (X-sets) can be detoxified with MEA to the extent necessary for shipment for ultimate disposal. The mustard concentration in the (Continued on reverse side)		

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20. **ABSTRACT (Contd)**

MF-A after the 8 days was approximately 0.6 ppm. The resulting solution was classified via DOT test as a corrosive liquid, less than a class B poison.

NOTE: DOT Department of Transportation.

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PREFACE

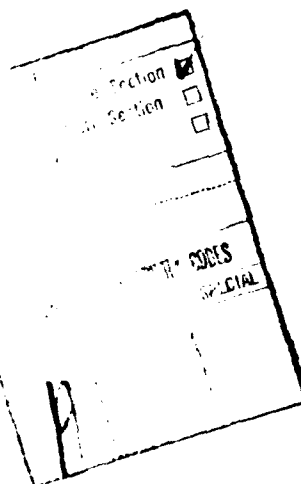
The work described in this report was authorized under the Project Manager for Chemical Demilitarization and Installation Restoration, Project No. 300, to investigate the detoxification of Navy sets (X-sets) and K955 sets. This work consisted of a 3-month effort from April 1976 to June 1976.

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DETOXIFICATION OF WAR GAS IDENTIFICATION SETS CONTAINING CHARCOAL IMPREGNATED WITH CHEMICAL AGENT

I. INTRODUCTION.

A. Background.

Disposal Engineering Division has been tasked with the disposal of obsolete war gas identification sets at Johnston Island. Included with these sets will be approximately 94 Navy sets (81 X-sets and 13 K955 sets) which consist of agent impregnated or adsorbed on charcoal which are planned to be collocated to Johnston Island from other OCONUS sites.

The X-sets are contained in a wooden box with a hinged cover. The box [7-1/2 wide by 16 long by 11-3/4 inches high (190.5 by 406.4 by 298.5 mm)] is divided into two compartments. In each compartment is a can 4 inches (101.6 mm) in diameter and 4-5/8 inches (117.5 mm) high surrounded by packing material. Inside each can is a bottle with a ground glass top. In the X302 set, one bottle contains nitrogen mustard (HN-1) and the other contains nitrogen mustard (HN 2). Both agents are adsorbed in 3 fluid ounces (90 cc) of activated charcoal. In the sample replacement sets there are two bottles in each set, but there is only one agent per set. The agents are as follows:

Set

X545 CG-simulant (contains triphosgene)

X546 CN (tear gas - chloroacetophenone)

X547 HS (mustard gas - H)

X548 L (lewisite)

X549 DM (adamsite)

X550 HN-1 (nitrogen mustard)

X551 HN-3 (nitrogen mustard)

X552 PS (chloropicrin)

The K955 sets are contained in a wooden box with a hinged cover. The box [15-1/2 wide by 30-3/8 long by 11-3/4 inches high (393.7 by 771.5 by 298.5 mm)] is divided into eight sections. Seven of the sections contain cans approximately 2 inches (50.8 mm) in diameter by 4-7/8 inches (123.8 mm) high. The cans are painted olive drab. The eighth section is empty. Each can contains a ground-glass stoppered bottle. Four of the bottles contain 3 fluid ounces (90 cc) of activated charcoal on which agent is adsorbed. One bottle contains lewisite (L); one contains chloropicrin (PS); and two contain mustard (H). A fifth bottle contains solid CG simulant (triphosgene).

The sets with HS (H), L, HN-1, HN-3, and PS each contain 0.85 fluid ounces (0.025 liters) of agent per bottle adsorbed on 3 fluid ounces (90 cc) of activated charcoal. The CG-simulant bottles each contain 3 grams of solid triphosgene. The bottles containing CG-simulant will be disposed of via the chemical neutralization reactor with caustic solution at Johnston Island as they contain no charcoal. The CN and DM are not classified as lethal agents and are not to be demilitarized during the operation at Johnston Island.

Prior to this work, a study was initiated by Chemical Laboratory, Edgewood Arsenal to investigate the reaction of mustard adsorbed on charcoal with monoethanolamine (MEA). It was concluded that the reaction between mustard adsorbed on charcoal and MEA did not proceed in accordance with pseudo first-order reaction kinetics and, under the conditions of this test, all of the agent could not be removed from the charcoal by chemical neutralization. A more detailed description of this work is in appendix A. Based on this study, an approach to detoxify the materials so that they would be suitable for compliance with Department of Transportation (DOT) guidelines for transportation to a site for ultimate disposal was undertaken.

B. Objective.

The objective of this pilot-scale test was to demonstrate that the X-sets and the K955 sets could be detoxified with MEA to the extent that the resulting products will pass a DOT test per CFR 21 and 49, so that the residue may be shipped for ultimate disposal as a class B or less poison.

II. EXPERIMENTATION.

This work was performed at APG-Edgewood Area (building E5625 Pilot Plant - Laboratory II).

A. Equipment.

1. Facilities.

This work was performed in an area approved for toxic work. This laboratory was ventilated by means of a 5000 ft³/min exhaust fan which draws air through a C23R1 charcoal and particulate filter before discharging into the atmosphere. All liquid drainage was handled through the 4000-gallon capacity toxic sump. This sump operates with an initial head of caustic solution, ensuring the pH to always be greater than 12. The sump has a separate ventilation system which is in operation whenever the laboratory (filling plant) is occupied. Whenever the sump reaches 80% capacity, the liquid waste is transferred to a 10,000-gallon direct displacement tank. The sump discharge will ultimately pass completely through the 10,000-gallon effluent tank, then through a pH neutralization process prior to discharge into the sanitary sewer.

2. Items of Equipment.

a. Drum, stainless steel, 15-gallon capacity with single 2-inch-diameter bung. The drum was fitted with temperature and pressure gauges as shown in figure 1. The bung was fitted with a 1/2-inch nipple, ball valve, and M12 filter as shown in figure 2.

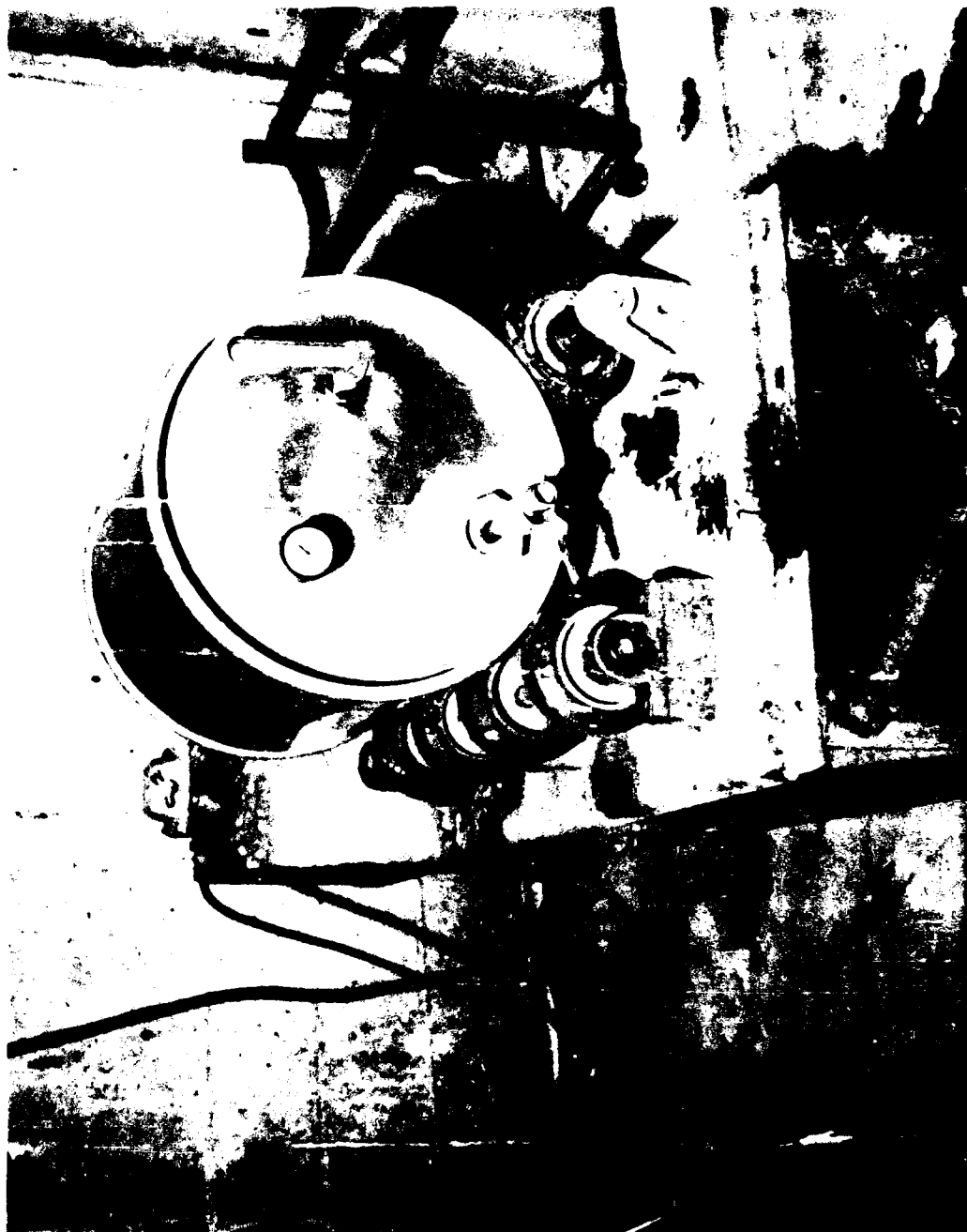


Figure 1 15-Gallon Stainless Steel Drum Fitted with
Temperature and Pressure Gauges



Figure 2. M12 Filter in Place on 15-Gallon Drum

- b. Hopper core sampler, 1-inch-diameter.
- c. Ball mill, 8-1/2-inch diameter, 1-gallon capacity, electrically driven.
- d. Roller, drum, adjustable, electrically driven.

An X-set, along with the test equipment, is shown in figure 3, less ball mill and core sampler.

B. Description of Operation.

The charcoal impregnated with agent from the Navy sets was added to a 15-gallon stainless steel drum which contained 6 gallons of MEA as shown in figure 4. The amount of agent added is shown in table 1. The mix was in the same proportion as would be used at Johnston Island. After the agent was added to the MEA, 3.4 gallons of additional MEA was added to the drum (total 9.4 gallons MEA). The drum was then sealed and rolled for 1 hour on the drum roller.

The drum was then allowed to sit for 6 hours and was then rolled again for 1 hour. The drum was rolled for 1 hour at approximately 9:00 a.m. and 2:00 p.m., Monday through Friday. No rolling took place on the weekends.

Each day after the first rolling, 100-ml samples were taken. No samples were taken on the weekends. These samples were analyzed for HD, based on the reaction rates for the agents (in neat form) involved. Mustard gas is the slowest reacting agent with MEA (see appendix A). The samples were also analyzed for PS to provide backup information on the slowest reacting agent.

When the MEA solution showed less than 5 ppm HS, 1 gallon of MEA/charcoal (after stirring) was ground in the ball mill. The ball mill was run until the proper particle-size range was reached. This range was 2 to 5 microns. This was the requirement for the DOT test that was conducted by the Environmental Toxicology Branch of the Biomedical Laboratory. The milled material, along with an MEA sample from the drum, was tested in accordance with DOT standard, Code of Federal Regulations, Titles 21 and 49. At the same time, a core sample of charcoal was tested for the amount of HS present. The operational portion of the Standing Operating Procedure is included in appendix B.

The material (MEA/charcoal) was held in the drum after the end of the test. Two samples were taken after the test ended, at 43 days and 149 days, to obtain long-term data. The drum had not been rolled since the eighth day of the test.

III. RESULTS.

A. Test and Analytical Data.

The pilot detoxification test data which were collected during this test is shown in table 2. Table 3 contains the analytic results from the daily samples.

The analytic data on table 3 show the sharp decline in the concentration of mustard in the MEA with respect to time. A graphic representation of the analytic data in table 3 is shown in figure 5.

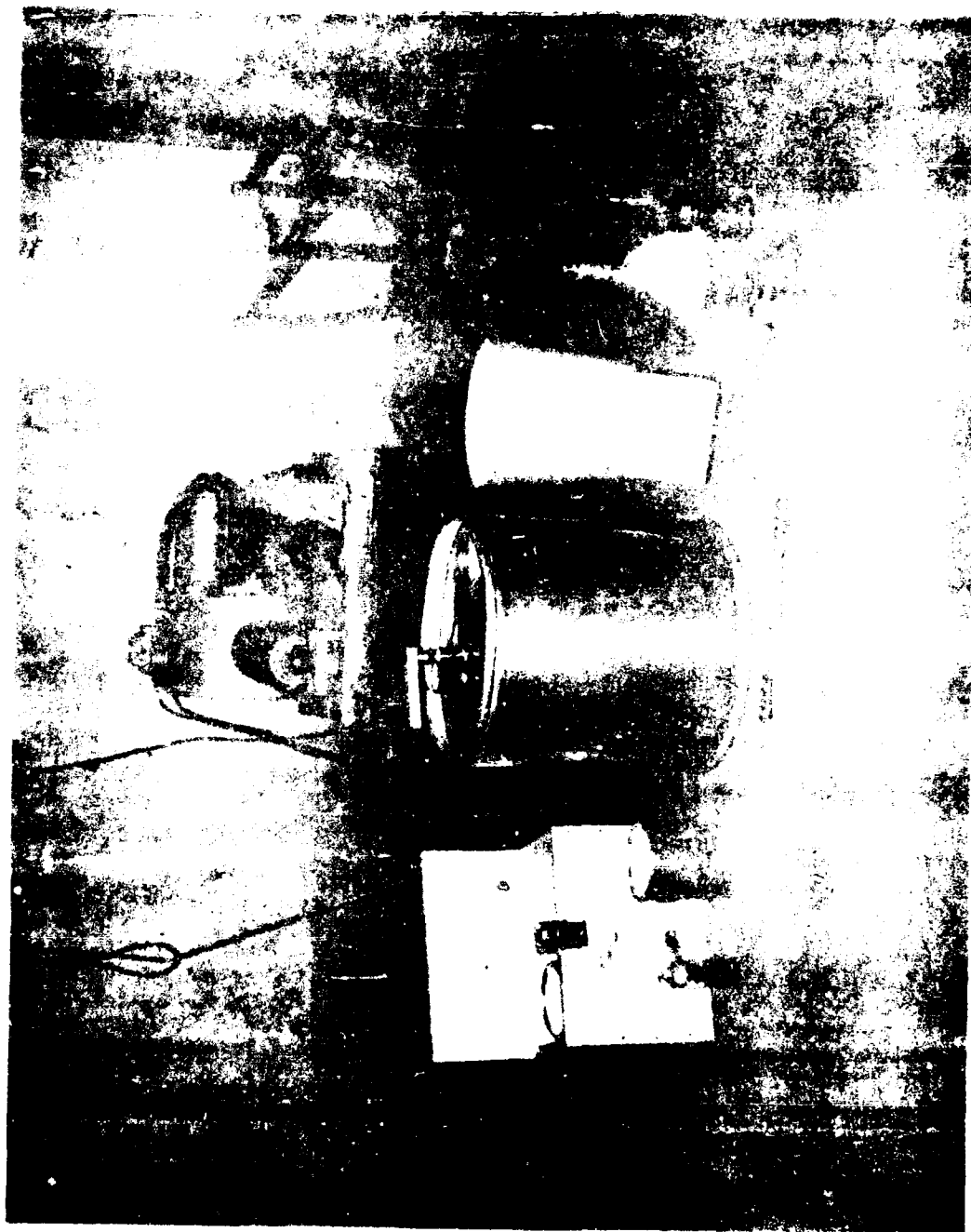


Figure 3. Fourt Xest Along with Test Equipment
(Ball Mill and C-10 Sampler Missing)



Figure 4 Addition of Set Contents to the Reaction Drum

Table 1. Chemical Neutralization of X-Sets

X-Set Johnston Island ^a				Edgewood Arsenal test		
Agent	Bottles	Charcoal ^b	Agent ^c	Bottles	Charcoal	Agent
		gm	ml		gm	ml
HS	66	2,970	1,650	8	360	200
PS	33	1,485	825	4	180	100
L	33	1,485	825	4	180	100
HN-1	78	3,510	1,950	9	405	225
HN-3	46	2,070	1,150	6	270	150
Total	256	11,520	6,400	31	1,395	775
		8 gal charcoal/agent 80 gal MEA ^d			0.94 gal charcoal/agent 9.4 gal MEA ^d	

^a Estimated Johnston Island inventory as of May 1976.

^b Based on 90 ml at .5 gm/ml.

^c Based on 25 ml per bottle.

^d Test will include a ratio of 10:1 MEA to agent and charcoal.

Table 2 Navy Set Pilot Test Data

Date	Time drum starts rolling	Drum pressure	Drum temperature	Ambient temperature	Time sample taken
		psi	°C	°C	
1 June 1976	1015	0	28	28	Sample No. 1 - 1127
	1035	0	28	28	
	1055	0	28	28	
	1115	0	28	28	
	1430	0	26	27	
	1450	0	27	27	
	1510	0	27	27	
	1530	0	27	27	
2 June 1976	0815	0	26	26	Sample No. 2 - 0935
	0835	0	26	26	
	0855	0	26	26	
	0915	0	26	26	
	0930	0	26	26	
	1400	0	26	26	
	1420	0	26	26	
	1440	0	26	26	
3 June 1976	1500	0	26	26	Sample No. 3 - 0900
	0745	0	26	26	
	0805	0	26	26	
	0825	0	26	26	
	0845	0	24	26	
	1400	0	26	26	
	1420	0	26	26	
	1440	0	26	26	
4 June 1976	1500	0	26	26	Sample No. 4 - 0915
	0800	0	24	24	
	0820	0	24	24	
	0840	0	24	24	
	0900	0	24	24	
	1430	0	23	23	
	1450	0	23	23	
	1510	0	23	23	
5 June 1976	1530	0	23	23	Sample No. 5 - 0910
	0800	0	25	25	
	0820	0	25	25	
	0840	0	25	25	
	0900	0	25	25	
	1400	0	25	25	
	1420	0	25	25	
	1440	0	25	25	
8 June 1976	1500	0	25	25	Sample No. 6 - 0935
	0820	0	25	25	
	0840	0	25	25	
	0900	0	25	25	
	0920	0	25	25	

Table 3. Analytic Data

Sample number and time sample taken	HS	Average concentration HS in MEA
	ppm	ppm
1	501.5	
11:15 1 June 1976	328.3	389.6
(1 hour)	339.1	
2	56.8	
	38.9	44.9
09:35 2 June 1976	38.9	
(23.35 hours)		
3	10.8	
	10.5	10.6
09:00 3 June 1976	10.3	
(45.75 hours)		
4	4.23	
	4.13	4.19
09:15 4 June 1976	4.19	
(70 hours)		
5	1.28	
	1.28	1.37
09:10 7 June 1976	1.64	
(142 hours)		
6	0.5	
	0.68	0.6
09:35 8 June 1976	0.63	
(166.25 hours)		

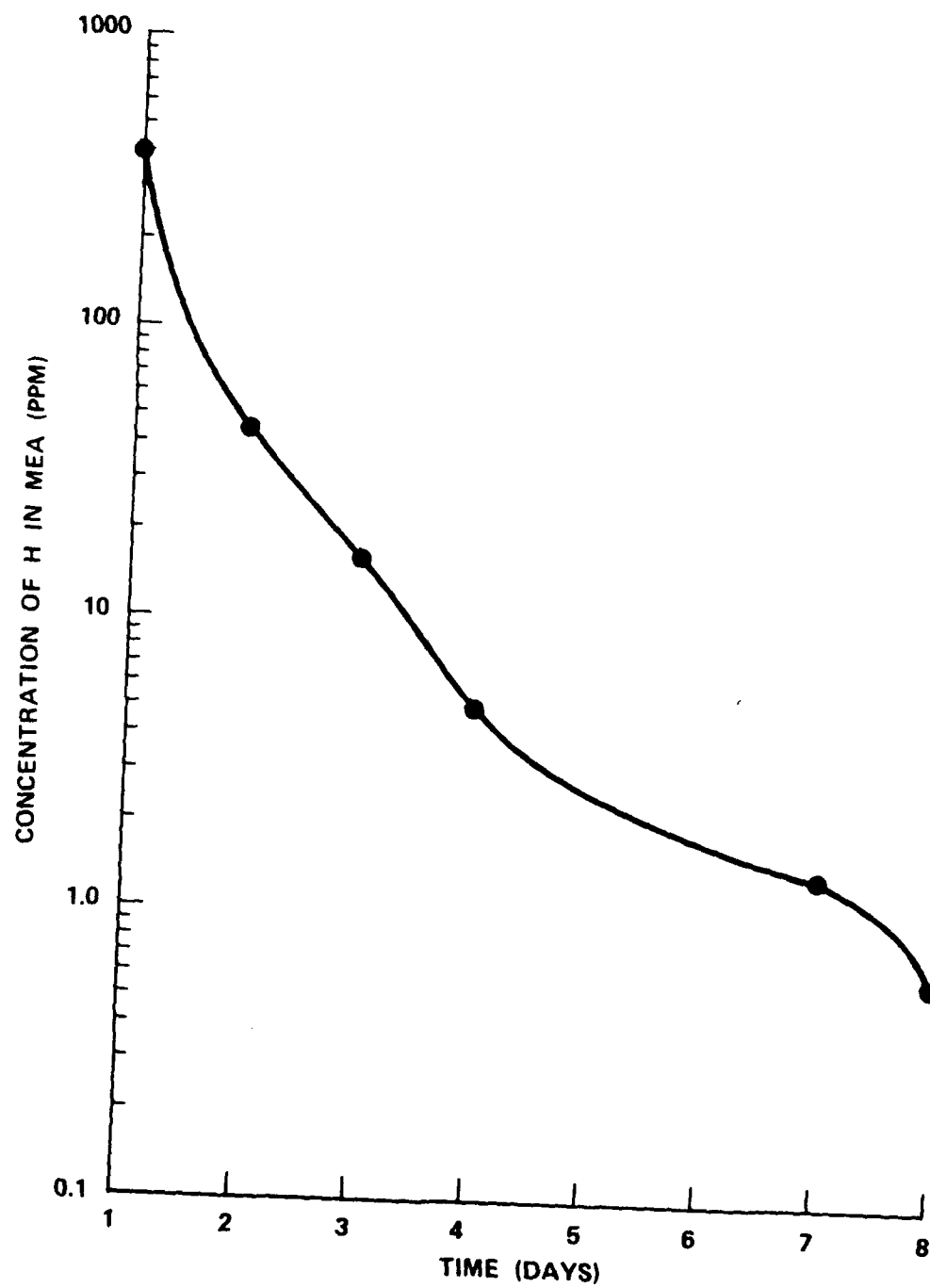


Figure 5. Mustard Concentration in MEA Versus Time

The daily MEA samples which were tested for mustard were also tested for PS. The detection limit for PS in the MEA solution was 10 ppm PS. All MEA samples showed less than 10 ppm. (The estimated detoxification time for PS is 3 hours at 34°C; it is a fast reaction compared to the mustard-MEA reaction.)

A sample of "decontaminated" charcoal was taken on the last day of the test and was analyzed for its mustard content. The amount of mustard present in 40.7 grams of MEA-saturated charcoal residue was determined to be 1.02 mg. This result was attained by three chloroform extractions and by heating the charcoal to a final temperature of 200°C and a vacuum of 10^{-5} torr Hg and then condensing out the residual mustard in a trap. A full description of the analytic procedures may be found in appendix A as performed by Chemical Laboratory in their report of 28 September 1976.

A sample of MEA was taken after 43 days of contact with Navy set contents and tested for mustard. The result of that test showed no mustard. However, a material was observed which had a retention time on the gas chromatograph (GC) similar to that of mustard and which was present in a quantity of less than 0.1 ppm.

A sample of MEA/charcoal taken after 149 days showed no mustard in the MEA or in the charcoal.

B. DOT Test.

Toxicity Tests.

Toxicology Division, Biomedical Laboratory, performed the required DOT tests per CFR 21 and CFR 49 and determined that the MEA solution, without the charcoal resulting from the detoxification of X-sets and K955 sets, passed the test and can be shipped as a corrosive liquid as less than a class B poison. In another sample, they also determined that the MEA solution with the charcoal passed the test and can be shipped as a corrosive liquid. (See appendix C.)

IV. DISCUSSION.

The test data presented in table 2 indicate that the reaction of the Navy sets and the replacement sets (X-sets) with MEA at ambient temperature is not an exothermic reaction. Therefore, there is no need for cooling of the reaction drum such as when chloroform is present. Although no pressure increase was noted, there was a very slight pressure increase which did not register on the gauge but was heard the first time the drum was opened.

The analytic data in table 3 has been shown graphically in figure 5 to show the sharp decline of mustard in MEA. In 8 days, the mustard concentration had dropped from an average concentration of 389.6 ppm to 0.6 ppm. A sample taken after 43 days showed no mustard. Another sample was taken after 149 days and showed no mustard in the MEA. (The detection limit for this test was 0.1 ppm mustard.)

The remaining charcoal showed 1.02 mg mustard recovered from 40.7 grams MEA saturated charcoal. This number cannot be related to the total quantity of mustard remaining in the charcoal because the 40.7 grams represent MEA-saturated charcoal, not dry charcoal, and the amount of MEA present was unknown. However, the amount of MEA was estimated and the total quantity of H in the final solution was then estimated to be approximately 2 ppm. The sample taken after 149 days showed no mustard in the charcoal. Further attempts to identify residual H on charcoal are indicated in appendix D.

The DOT tests showed the material to be a corrosive liquid, less than a class B poison. This would facilitate shipment to a CONUS site for ultimate disposal.

The overall results of this test indicate that the process works and should be considered to be a viable option for the detoxification of Navy and X-sets. A full-scale procedure, for the detoxification of Navy and X-sets expected to be located at Johnston Island is included in Appendix E.

V. CONCLUSIONS.

It is concluded that the Navy sets to be collocated to Johnston Island can be detoxified and shipped back to CONUS as a corrosive liquid for ultimate disposal.

This technique could also be applied to the movement of other Navy sets in CONUS, if necessary.

APPENDIX A

DECONTAMINATION WITH MONOETHANOLAMINE OF H-IMPREGNATED CHARCOAL CONTAINED IN OBSOLETE NAVY WAR GAS IDENTIFICATION SETS

Chemical Laboratory

28 September 1976

I. INTRODUCTION.

At the request of Edgewood Arsenal Demilitarization/Disposal Officer personnel, this study was initiated to ascertain the feasibility of decontaminating agent-impregnated charcoal contained in obsolete Navy war gas identification sets with monoethanolamine (MEA). These sets were prepared, circa 1940, by separately placing 25-ml amounts of H, HN-1, HN-3, L, or PS in ground-glass stoppered bottles containing 90 ml of charcoal. No information was available concerning either the original purity of the various agents, or the type, source, bulk density, or internal pore area of the carbon used in the preparation of these sets. Work conducted in this study was primarily limited to the decontamination of H-impregnated charcoal because of funding and time limitations. The H-impregnated charcoal system was selected for study in preference to the other agent-charcoal systems contained in these sets since Environmental Research Division personnel are of the opinion that the H-charcoal system would probably be the most difficult system to decontaminate with MEA under the initially proposed field decontamination procedure of using a 10:1 v/v ratio of MEA/agent.

The general approach followed in this study consisted of three discrete but interrelated parts. Part I consisted of an examination of charcoal (of a known mesh size, bulk density, and internal pore area) impregnated with H and subsequently decontaminated with MEA. Part II consisted of an examination of the decontamination of actual samples of H-impregnated charcoal contained in the Navy sets. Part III consisted of an examination of a pilot plant scale process involving the collective decontamination in MEA of Navy set charcoal samples impregnated with agents H, HN-1, HN-3, L, and PS.

II. RESULTS AND DISCUSSION.

Part I - Pittsburgh Activated Carbon Study.

In this work, two samples of Pittsburgh Activated Carbon (PAC), BPL grade, lot 6098, charcoal, each weighing approximately 8.2 grams, were impregnated with 5 ml (approximately 6.2 grams) of H and allowed to reach sorption equilibrium over a period of approximately 3 weeks at room temperature. The PAC charcoal represented a well characterized charcoal with a nominal US sieve range of 12-30 mesh, a bulk density of 0.459 gm/ml, and an internal pore area of 1058 m²/gm. The ratio of H to charcoal used in the preparation of these two samples was equivalent to that used in the original preparation of the Navy sets.

In an effort to determine the amount of H irreversibly sorbed on the PAC charcoal, one of the impregnated samples described above was placed in a specially constructed high vacuum system. This system was equipped with provisions for heating the sample in 10°C increments over a temperature range from ambient (25°C) to 200°C and for collecting any distilled H at liquid nitrogen temperatures in a glass trap. This system was operated under these conditions at a pressure of 10^{-5} torr. The sample of H-impregnated charcoal was degassed over a period of 15 workdays. H removed from the charcoal was collected and weighed for two sample temperature increments; 25° to 100° C and 100° to 200°C. The H content of each fraction was determined by gas liquid chromatography (GLC) analysis. The first fraction (25° to 100°C) was determined to be 98.2% H, which compared favorably to an initial H purity of 99.6%. The second fraction was determined to have a purity of 77.9%, indicating some thermal degradation of the H. The material collected in the first fraction amounted to 49.5% of the original amount of H placed on the charcoal, and the combined weight of both fractions accounted for 89.8% of the original amount of H used. Comparison of the initial weight of the charcoal, before impregnation with H, with the weight of the impregnated sample after degassing indicated that 5.1% of the H remained on the charcoal either as polymerized or physically and/or chemically bound H. Further analysis of data indicated that an additional 5.1% of the original weight of H placed on the charcoal could not be accounted for and was either condensed elsewhere in the vacuum system or simply escaped from the vacuum system over the prolonged degassing period. The most important information derived from this experiment, however, was that even under these stringent degassing conditions, approximately 5% of the original amount of H placed on the charcoal could not be removed from within the charcoal matrix.

In the original plans established for the conduct of this study, experimental work, complementary to the work described above, was planned. This work was designed to establish the extraction efficiency of chloroform for H impregnated on well characterized charcoal, i.e., one with a known bulk density, particle size, and internal pore surface area. Manufacturing Technology Directorate and Demilitarization/Disposal Office personnel directed that these investigations be abandoned, and an abbreviated experimental program was subsequently conducted.

The second sample of H-impregnated PAC charcoal, referred to above, was placed in an Erlenmeyer flask to which 50 ml of MEA was added to produce a 10:1 v/v MEA/H ratio. The mixture was stirred for 2-1/2 hours at 50°C and then was allowed to remain in an unstirred condition at room temperature, 25°C, over a 48-hour period. This sample was subsequently stirred for an additional 6-hour period. During this reaction period, the concentration of the H in the MEA should have been reduced to approximately 1 ppm assuming that the homogeneous pseudo first order of the H with the MEA and that all the H put on the carbon was immediately available for reaction with the MEA. After this last stirring period, the MEA was separated from the charcoal and tested for the presence of H. This test involved placing 10 ml of the MEA reaction mixture in a separatory funnel and adding 50 ml of an aqueous 10% NaCl solution followed by 1 ml of hexane. This mixture was shaken vigorously, and the organic and aqueous phases were allowed to separate. The hexane was then removed and tested for the presence of H by gas chromatography. The extraction efficiency of H into hexane under the conditions outlined above was previously determined to be virtually 100%. Gas liquid chromatography analysis indicated that less than 2 ppm of H (minimum detectable limit) was present in the MEA separated from the PAC charcoal. The charcoal itself was subsequently extracted at three separate times with 50 ml aliquots of chloroform, a mutual

solvent for both H and MEA. Direct GLC analysis of these three extracts indicated H concentrations of 239, 154, and 80 ppm, respectively. These data are summarized in table A-1.

Table A-1. H Content of Chloroform Extracts of Decontaminated PAC Carbon

Chloroform extract*	H concentration**	Weight equivalent of H	Amount of original H
	ppm	gm	%
1	239	0.016	0.25
2	153	0.010	0.16
3	80	0.005	0.08
Total H in chloroform extracts		0.031	0.49 ± 0.11

* With stirring, the chloroform remained in contact with the carbon for a 1-hour period for the first extract, 45 minutes for the second extract, and 16 hours for the third extract.

** Analysis was done with a HP5830 gas chromatograph equipped with flame ionization detectors. This column was 6 foot by 1/8 inch stainless steel packed with 10% UCW98 on 80/100 HP (AWDMCS) chromosorb W. Conditions were: oven temperature - 155°C isothermal, carrier flow - 45 ml/min, injection port temperature - 250°C, and flame ionization detectors temperature - 300°C.

Although no detectable amount of H was found in the separated MEA phase, the data in table A-1 indicate that at least 0.5% of the original H placed on the charcoal was not destroyed during the decontamination process. These data appear to indicate that, at least for some of the H contained in the internal pore structure of the charcoal, the decontamination process was affected by the diffusion of H from within the internal pore structure of the charcoal into the MEA.

Part II - Decontamination of Navy Set Charcoal.

In an effort to corroborate the experimental findings obtained from the PAC charcoal work, an entire unit of H-impregnated charcoal from the actual Navy war gas identification set (reportedly 25 ml of H on 90 ml of charcoal) was decontaminated with 250 ml of MEA. An entire unit was used to preclude the possibility of errors introduced by the possibility of unequal sorption of the H by the charcoal or non-representative sampling of the charcoal. The mixture was stirred for 4-3/4 hours at 50°C; stirring was continued at 25°C for 19 additional hours. Assuming that the homogeneous pseudo first-order reaction of H with the MEA and that the 25 ml of agent on the charcoal was absolutely pure, the time of reaction of the MEA with the H was sufficient to reduce the concentration of H in the MEA to below 1 ppm. In accordance with previously established procedures, the MEA was separated from the charcoal and extracted with hexane. The H content of the MEA was determined by GLC analysis of the hexane extract. This analysis indicated that an H concentration of 40 ppm remained in the MEA. The carbon was sequentially extracted with

chloroform three times. Gas liquid chromatography analysis of these extracts indicated the presence of 230, 400, and 755 ppm concentrations of H in the first, second, and third chloroform extracts, respectively. These data, along with that for the MEA decontaminant, are summarized in table A-2.

Table A-2. Gas Liquid Chromatography Analysis Data for H Concentration in Products from the Decontamination of Navy Set Charcoal with MEA (Trial 1)

Material analyzed	H concentration*	Amount of H	Amount of original H
	ppm	gm	%
MEA	40	0.013	0.04
1st CHCl ₃ extract**	230	0.030	0.09
2d CHCl ₃ extract**	400	0.052	0.16
3d CHCl ₃ extract**	755	0.098	0.30
Total amount of H recovered		0.193	0.59 ± 0.06

* Gas liquid chromatography conditions used in this work, with minor exceptions, were essentially the same as those stated in table A-1.

** The three successive extractions were each done using 100-ml volumes of CHCl₃. In each extraction, the CHCl₃ remained in contact with the carbon, with stirring, at 25°C, for a period of 1 hour.

Examination of the data in table A-2 indicates that, as with the PAC charcoal, the reaction of at least some of the H on the Navy set charcoal with the MEA is affected by the diffusion of H from within the internal pore structure of the charcoal into the MEA.

To confirm these observations, a second unit of H-impregnated charcoal from the Navy sets was decontaminated in a manner similar to the first. The second unit was immersed in 250 ml of MEA at 50°C for 64 hours. Because of a stirrer malfunction sometime during this period, the stirring time of mixture is not known. The mixture, however, is known to have received an initial period of stirring of at least 1 hour. To preclude the possibility of insufficient reaction time the mixture was then reacted at 50°C with stirring for a period of 7 hours. Again assuming the homogeneous, pseudo first-order reaction of H with MEA, 100% agent purity and homogeneity of the reactions, the concentration of residual H in the MEA should have been well below 1 ppm. In accordance with previously established procedures, the MEA was determined by GLC analysis of the hexane extract. Three successive extractions of the charcoal with chloroform were also made and the extracts assayed by GLC for H content. After the third extraction, a portion of the charcoal residue was placed in the vacuum system and degassed for a period of several days at a pressure of 10⁻⁵ torr. The carbon was gradually heated to 200° in 10°C increments. The amount of H in the chloroform condensate, collected at liquid nitrogen temperature, was assayed by GLC. The results of these analyses are presented in table A-3.

Table A-3. Concentration of Residual Agent in Reaction Products From the Decontamination of H-Impregnated Carbon in Navy War Gas Identification Sets With MEA (Trial 2)

Materials analyzed ^a	H concentration	Amount of H	Amount of original H
	ppm	gm	%
MEA	<1		
1st CHCl ₃ extract ^b	<1		
2d CHCl ₃ extract ^b	135	0.009	0.028
3d CHCl ₃ extract ^b	120	0.016	0.049
CHCl ₃ vacuum distillate	29	0.001	0.003 ^c
Total amount of H recovered		0.026	0.080 ± 0.003

^a All material analyzed by GLC. Gas liquid chromatography conditions used, with minor exceptions, were essentially the same as those stated in table A-1.

^b Fifty liter of CHCl₃ were used for each of the first and second extractions, while 100 ml of CHCl₃ were used for the the third extraction. In all extractions, the CHCl₃ remained in contact with the carbon, with stirring, for a period of 1 hour at 25°C.

^c This percentage represents the normalized amount of residual agent to be found after MEA decontamination of a single unit of H-impregnated charcoal contained in the Navy sets (90-ml charcoal with 25 ml of H). This normalization is based on the weight of the degassed charcoal (5.3492 gm), an assumed charcoal bulk density of 0.459 gm/ml, the amount of CHCl₃ solution containing H collected in the liquid nitrogen trap (6.5525 gm), the density of CHCl₃ at 25°C (1.47 gm/ml), and an average assumed density for H of 1.27 gm/ml.

Examination of the data in table A-3 again indicates that the decontamination of H on the Navy sets charcoal is not complete. It would again appear that the reaction of at least some of the H on the Navy sets charcoal with MEA is affected by diffusion of H from within the internal pore structure of the charcoal into the MEA.

Part III of this study consisted of a pilot scale test, conducted by Manufacturing Technology Directorate personnel in which Navy set charcoal contaminated with several agents, was placed in a steel drum containing 9.4 gallons (34.6 liters) of the decontaminant, MEA. The quantity of each agent used in this test is shown in table A-4.

Table A-4. Type and Amount of Agents Used in Pilot Scale Decontamination of Agents in Navy Identification Sets

Item	Agent	Number of set	Volume of each	Volume of charcoal
			liters	liters
1	HD	8	.200	.720
2	PS	4	.100	.360
3	L	4	.100	.360
4	HN-1	9	.225	.810
5	HN-3	6	.150	.540
Total			.775	2.790

The ratio of MEA decontaminant to total agent used was 46 to 1. This test was conducted at ambient temperature (24° to 27°C). The reaction components were mixed by rotating the drum on a drum roller for 1 hour each morning and afternoon during the testing period. A sample of MEA was periodically withdrawn from the drum after the first mixing period. This sample was extracted with chloroform immediately upon receipt and the extract was subsequently analyzed for the presence of H and PS. Extraction and CLC procedures used were similar to those described previously in section II, part 1, of this report. The MEA was tested for the presence of H on the basis of the rationale presented in the addendum to this appendix in which it was indicated that H would probably be the slowest reacting agent of any of the agents present in the MEA decontaminant. At the request of Edgewood Arsenal, Demilitarization Disposal Office personnel, the solutions were also tested for the presence of PS, since this material was expected to be one of the most reactive of the agents present in the decontamination solution. A summary of the GLC analysis performed on samples of the MEA decontaminant taken at successive time intervals is presented in table A-5.

Table A-5. Gas Liquid Chromatography Analysis of MEA Decontamination Solution
From Pilot Plant Demilitarization of Navy Sets

Sample number	Date received ^a	Intervals after start of decontamination process	H content in MEA ^{b,c}
		hr	ppm
1	1 June 1976	1	390 ± 97
2	2 June 1976	23	45 ± 10
3	3 June 1976	46	11 ± 6
4	4 June 1976	70	4.2 ± 0.3
5	7 June 1976	142	1.4 ± 0.5
6	8 June 1976	166	0.6 ± 0.3
7	13 July 1976	1008	≤ 0.1 (lower detectable limit)

^a All samples were extracted 10:1 v/v CHCl₃/MEA immediately upon receipt from the pilot plant with the exception of sample number 7, which was extracted on 13 July 1976.

^b Values reported represent average values obtained from three individual determinations. Extraction procedures and GLC conditions are the same as those indicated earlier in this report.

^c Samples 1, 2, 4, 5, and 6 were also analyzed for the presence of PS. All samples tested indicated concentrations of PS of less than 10 ppm.

Examination of the data presented in table A-5 again indicates that the decontamination of the H on the charcoal with MEA is affected by diffusion of H from within the internal pore structure of the charcoal into the MEA.

In the GLC analysis of the MEA decontamination product solution, interferences caused by the presence of unidentified components precluded the detection of PS in the product solution at concentrations of less than 10 ppm. Since the minimum detectable limit for PS in the PS-MEA reaction product solutions had been previously established at less than 1 ppm, this interference was attributed to the presence of either reaction products formed during the decontamination of the other agents present in the MEA or, possibly, to the presence of agent-agent interaction products. Several attempts were made to improve the detection sensitivity of the PS in the MEA product solution by altering various gas chromatographic operating conditions and using different column substrate materials. All attempts to accomplish this proved unsuccessful, and experimental results indicated that a more extensive investigation would be required to attain the desired objective. Such a program was considered to be outside the scope of the present work, and further attempts to resolve this problem were abandoned. It should be noted, however, that none of the solutions tested indicated concentrations of PS greater than 10 ppm.

In consonance with the experimental work described above, a sample of carbon residue was removed from the reaction drum after a reaction period of 166 hours. This sample was extracted with chloroform and was subsequently vacuum distilled in accordance with procedures described in section II, parts I and II, of this report. The chloroform extracts and collected distillate were analyzed for the presence of residual H. The results of these analyses are presented in table A-6.

Table A-6. Analysis of Charcoal Residue^a From Pilot Plant
Demilitarization of Navy Sets

Item	Amount of H recovered
	mg
Chloroform extract No. 1	0.36
Chloroform extract No. 2	<0.05
Chloroform extract No. 3	0.09
Degassed charcoal ^b	0.57 ^c

^a Sample received on 11 June 1976. This sample of MEA saturated charcoal residue (40.7 gm) was extracted three times successively with 50-ml portions of chloroform. During each extraction the charcoal was stirred in the chloroform for a period of 1 hour. The chloroform extract was then analyzed for H by the GLC method described previously in this report.

^b Following the third extraction, a portion of the charcoal residue was placed in a vacuum apparatus and degassed at a pressure of 10^{-5} torr for a period of 16 hours at a sample temperature ranging from 25° to 200°C. Degassed vapor from the charcoal was collected in a liquid nitrogen trap and subsequently analyzed for H content by the GLC method previously described in this report. The weight of the degassed carbon was 6.9433 gm.

^c The amount of residual H shown has been normalized to represent the total amount of H that would be degassed from the entire 40.7 gm charcoal sample.

Analysis of the data in table A-6 indicates that the decontamination of the H on the Navy set charcoal with MEA is affected by the diffusion of H from within the internal pore structure of the charcoal into the MEA.

III. CONCLUSIONS.

1. Approximately 5% of the H impregnated on PAC, type BPL, charcoal was apparently irreversibly sorbed on the charcoal.

2. Decontamination of H impregnated on PAC or Navy set charcoal with MEA is affected by the diffusion of H from within the internal pore structure of the charcoal into the MEA. Consequently, the time period required for the decontamination of H on charcoal with MEA cannot be predicted on the bases of reaction rate data previously established for the homogeneous, pseudo first-order reaction of H with excess MEA (1:5 v/v ratio of H to MEA).

3. In consonance with the above paragraph, charcoal samples obtained from a pilot plant process investigation involving the decontamination of agent impregnated Navy set charcoal with MEA (1.46 v/v ratio total agent to MEA) under ambient conditions of temperature (24° to 27°C) was found to contain residual H after a decontamination period of approximately 1 week (166 hours). Had the H on the charcoal been immediately extracted by this large excess of MEA, the amount of H remaining would have been undetectable by our methods of analysis.

4. Simple extraction of H from charcoal with a chloroform solvent does not appear to be a viable method for the quantitative removal of H impregnated on charcoal.

5. Because of the difficulty experienced in removal of the H from the charcoal by vacuum distillation at elevated temperature, it does not appear feasible to distill residual H from contaminated charcoal at atmospheric pressure.



ADDENDUM

DEPARTMENT OF THE ARMY
HEADQUARTERS EDGEWOOD ARSENAL
ABERDEEN PROVING GROUND MARYLAND 21010

SARFA-MT-F

10 June 1976

MEMORANDUM FOR RECORD

SUBJECT: Detoxification of Navy Sets

1. The following is a reply to a directed action from a meeting on the Identification sets on 26 May 1976, in which Edgewood Arsenal was directed to provide a rationale for the assumption that mustard is the slowest reacting agent in the Navy set with respect to the decontaminant, monoethanolamine (MEA).
2. Disposal Engineering Division has been tasked with the disposal of obsolete war gas identification sets at Johnston Island. Included with these sets are approximately 133 Navy sets (or X-sets) and approximately 13 K955 sets, which consist of agent impregnated on charcoal, which are planned to be collocated to Johnston Island from other OCONUS sites.

The Navy sets are contained in a wooden box with a hinged cover. The box measures 7-1/2 wide by 16 long by 11-3/4 inches high (19.1 by 40.6 by 29.8 cm) and is divided into two compartments. In each compartment is a can 4 inches (101.6 mm) in diameter and 4-5/8 inches (117.5 mm) high surrounded by packing material. Inside each can is a bottle with a ground glass top. In the X302 set, one bottle contains nitrogen mustard (HN-1), the other, nitrogen mustard (HN-3). Both agents are adsorbed on 3 fluid ounces (90 cc) of activated charcoal. In the sample replacement sets, there are two bottles in each set, but there is only one agent per set. The agent are as follows:

Set

X545 CG-simulant (contains triphosgene)
X546 CN (tear gas - chloroacetophenone)
X547 HS (mustard gas - H)
X548 L (lewisite)
X549 DM (adamsite)
X550 HN-1 (nitrogen mustard)
X551 HN-3 (nitrogen mustard)
X552 PS (chloropicrin)

The K955 sets are contained in a wooden box with a hinged cover. The box measures 15-1/2 wide by 30-3/8 long by 11-3/4 inches high (39.4 by 77.2 by 29.8 cm) and is divided into eight compartments. Seven compartments contain a can and bottle as described above. The K955 set contains the following agents:

One bottle L, one bottle PS, two bottles HS, one bottle CG-simulant, one bottle CN, and one bottle DM. The sets with HS, L, HN-1, HN-3, and PS each contain 1.7 fluid ounces (0.050 liters) of

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10 June 1976

SUBJECT: Detoxification of Navy Sets

agent, or 0.85 fluid ounce (0.025 liters) of agent per bottle on 90 cc of activated charcoal. The CG-simulant set contains 6 grams of triphosgene. The bottles containing CG-simulant will be disposed of via the chemical neutralization reactor with caustic solution as they contain no charcoal. The CN and DM bottles each contain approximately 5 grams of nonlethal solid agent. CN and DM are not classified as lethal agents and are not to be demilitarized during this operation.

3. An experimental plan has been initiated to decontaminate charcoal impregnated with agents HS, HN-1, HN-3, L, and PS with MEA to determine if the reaction products will pass the DOT test as less than class B poison. A 10:1 volume ratio of MEA/agent on charcoal will be used in all tests. It is believed that the reaction of MEA with HS on charcoal will be the slowest of the various agent/decontaminant reactions. This judgement is based on experimental information obtained from several research investigations conducted by Chemical Laboratory and from private communication with several Chemical Laboratory personnel.

4. The following is offered in support of this statement. The pseudo first-order reaction rates for MEA with neat HD, 10% v/v HN-1 in chloroform and 50% v/v PS in chloroform were determined by Chemical Laboratory in previous investigations concerning the demilitarization of agents contained in the K951-4 war gas identification sets. Kinetic data obtained for the reaction of agents HD and HN-1 with MEA are presented in tables 1 and 2, below.

Table 1. Kinetic Data for the MEA/HD Reactions*¹

Temperature	Half life (t _{1/2})	K _{obsd} min ⁻¹
°C	min	
25.2	321.0	2.16 × 10 ⁻³
35.2	82.0	8.4 × 10 ⁻³
44.4	39.6	1.75 × 10 ⁻²
57.0	11.0	6.06 × 10 ⁻²

* A 5:1 ratio of MEA/HD was used in all tests.

Table 2. Kinetic Data for the Reaction* of 10% v/v HN-1 in Chloroform with Monoethanolamine**2

Temperature	K_0 min^{-1}	$t_{1/2}$ min	Linear corrected efficient	Standard error in K_2
°C				%
25.1	0.0201	34.6	0.998	1.70
25.1	0.0200	34.7	0.9996	0.85
34.9	0.0675	10.3	0.999	1.59
34.9	0.0533	13.0	0.999	0.99
44.9	0.138	5.03	0.998	1.72
44.9	0.135	5.14	0.999	1.18

* All reactions followed through greater than 90% reaction.

** A 10:1 ratio of MEA: 10% v/v HN-1 in chloroform was used in all tests.

The pseudo first-order rate constant for the reaction of PS (50% v/v PS in chloroform) with MEA was determined³ to be:

$$K_{\text{obs}} = K' = 7.47 \times 10^{-2} \text{ min}^{-1}$$

The half-life of the reactions of PS with MEA under pseudo first-order conditions was calculated from this rate constant to be 9.3 minutes at 34°C. In another investigation concerning the decontamination of agent contaminated dunnage from the K951-4 war gas identification sets, it has been indicated⁴ that MEA could be an effective decontaminant for L, CK, and CG as well as for HD, HN-1, and PS. In that study, 5% L in chloroform was decontaminated with MEA using a 10:1 volume ratio of agent in chloroform to MEA. The decontaminated sample was sent to Biomedical Laboratory for toxicity testing and was found to pass the DOT tests as less than a class B poison. In a private communication⁵ with Dr. George Davis (SAREA-CL-DC), the principle investigator who studied the reaction, it was learned that the reaction of the L in chloroform with MEA appeared to be complete in a matter of seconds at room temperature. Dr. Davis further indicated that the actual rate of this reaction could not be accurately determined, since the rate of mixing of the two components appeared to be rate controlling. An investigation is presently being conducted by the Environmental Research Division of Chemical Laboratory to establish the rate of the reaction of a 1:10 volume ratio of

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5% v/v L in chloroform with MEA. Work will also be conducted to isolate and identify the products of this reaction. In the referenced conversation, Dr. Davis was also questioned concerning the rate of reaction of HN-3 with MEA. Dr. Davis indicated that, although the rate of this reaction had not been studied, its rate should be of the same order of magnitude as that for the reaction of HN-1 with MEA and should be considerably faster than the rate of reaction between sulfur mustard (HS) and MEA.

5. Based on the above information, it is concluded that MEA is an effective decontaminant for L as well as for HS, HN-1, HN-3, and PS, and that the slowest reacting agent is HS.

1 Incl
References

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CONCUR:

DR. JOSEPH EPSTEIN
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Chemical Laboratory

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SUBJECT: Detoxification of Navy Sets

10 June 1976

References

1. DF, SAREA-CL-PDS, Cml Lab, subject: Decontamination of Toxic War Gas Sets, 28 March 1974.
2. DF, SAREA-CL-DA, Cml Lab, subject: Decontamination of Toxic War Gas Sets, 16 December 1974.
3. DF, SAREA-CL-DP, Cml Lab, subject: Decontamination of Toxic War Gas Sets, 16 December 1974.
4. DF, SAREA-CL-D, Cml Lab, subject: Decontamination of Dunnage from the K941-2 and K951-4 Toxic War Gas Sets, 18 March 1975.
5. Private communication between Dr. George Davis (SAREA-CL-DC) and Mr. Steve Rosenberg (SAREA-MT-E) on 1 June 1976.

APPENDIX B

SOP - PILOT TEST FOR THE CHEMICAL NEUTRALIZATION OF NAVY WAR GAS IDENTIFICATION SETS (X-SETS)

Manufacturing Technology Directorate

24 May 1976

1. Brief Statement of Work to be Done.

This test is being conducted to demonstrate that the Navy war gas identification sets (Navy sets) can be detoxified by chemical neutralization. These Navy sets contain mustard gas (HS), lewisite (L), nitrogen mustard (HN-1), nitrogen mustard (HN-3), and chloropicrin absorbed on charcoal. Each set contains two bottles packed and sealed in separate 4-inch-diameter cans. The two cans are packed in separate compartments inside a wooden box (7-1/2 by 16 by 11-3/4 inches). In each bottle, 25 ml of agent are absorbed on 90 cubic centimeters of activated charcoal. A total of 31 bottles of agent-activated charcoal are to be neutralized using monoethanolamine (MEA). The distribution of agent to be detoxified is as follows:

<u>Agent</u>	<u>No. of bottles</u>	<u>Agent</u> ml	<u>Charcoal</u> gm
HS	8	200	360
PS	4	100	180
L	4	100	180
HN-1	9	225	405
HN-3	6	150	270
Total	31	775	1,395

The charcoal impregnated with agent will be added to 6 gallons of MEA in a 15-gallon stainless steel drum. After this addition, 3.4 gallons of MEA will be added to the drum. This drum will then be sealed, placed on a drum roller, and rolled for 1 hour. A sample of the drum solution will be taken and analyzed for HS. The drum will then be allowed to sit for 5 hours and then rolled again for 1 hour. This sequence will continue for each working day until results of the analyses of the MEA solution indicate less than 5 ppm HS. When this occurs, 1 gallon of the MEA/charcoal solution will be drawn off, ground in a ball mill to a particle size range of 2 to 5 microns, and tested by Biomedical Laboratory in accordance with DOT standards.

2. Equipment to be Used.

a. Facilities.

Work will be performed in an area approved for toxic work. This area, designated Lab II (Filling Plant), is located on the first floor of building E5625. This laboratory is ventilated by a 5000 ft³/min exhaust fan which draws air through a C23R1 charcoal and particulate filter before discharging it into the atmosphere. All liquid drainage will discharge into a toxic sump (capacity 4000 gallons). This sump operates with an initial head of caustic solution, ensuring that the pH will always be greater than 12. This sump has a separate ventilation system which is in operation at all times whenever the Filling Plant is occupied. From this sump, the liquid waste will be transferred to a 10,000-gallon direct displacement tank. (This occurs whenever the sump reaches 80% capacity.) Ultimately, the sump discharge will pass completely through the 10,000-gallon effluent tank, then through a pH neutralization process before it discharges into the sanitary sewer.

b. Items of Equipment.

- (1) Drum, stainless steel, 15-gallon with bung, 2-inch diameter, fitted with 1/2-inch nipple, ball valve, temperature and pressure gauges.
- (2) Hopper core sampler, 1 inch diameter.
- (3) Ball mill, 8-1/2 inch diameter, capacity 1 gallon.
- (4) Roller, drum, adjustable, electric powered.

3. Materials to be Present.

- a. HS (2,2'-dichloro-diethyl sulfide) (ClCH₂CH₂)₂, fp 14°C, bp 227.8°C, density 1.27 gm/ml @ 20°C, vapor pressure 0.072 mm Hg @ 20°C.
- b. HN-1 (2,2'-dichloro-triethylamine) (ClCH₂CH₂)₂NC₂H₅, fp -34°C, bp 85°C @ 10 mm Hg (decomposes at atmospheric pressure below 85°C), liquid density 1.24 @ 25°C, vapor density (compared to air) 5.9, vapor pressure 0.25 mm Hg @ 25°C.
- c. HN-3 (2,2',2''-trichloro-triethylamine) N(CH₂CH₂Cl)₃, fp -4°C, bp 137°C (decomposes at atmospheric pressure below 137°C), liquid density 1.24 @ 25°C, vapor density (compared to air) 6.9, vapor pressure 0.0109 mm Hg @ 25°C.
- d. L (dichloro(2-chloro-vinyl)arsenic) ClCH=CHAsCl₂, fp -18°C, bp 109°C, density 1.89 gm/ml @ 20°C, vapor pressure 0.394 mm Hg @ 20°C.
- e. PS (chloropicrin) CCINO₃, fp -64°C, bp 112°C, density 1.69 gm/ml vapor pressure 44 mm Hg @ 34°C.

f. MEA (monoethanolamine) $\text{NH}_2\text{CH}_2\text{CH}_2\text{OH}$, fp 10.5°C , bp 170.5°C , flash point 93°C , density 1.02 gm/ml @ 20°C , vapor pressure 6 mm Hg @ 60°C .

g. 5% HTH solution (5% high test hypochlorite-water solution) used for decontamination of spills.

h. Charcoal, activated, particle size range 10-30 mesh, used to absorb agents.

i. Acetylene, $\text{HC}\equiv\text{CH}$, fp -84°C , bp -81°C , flash point -18°C , density 1.17 gm/l, vapor pressure 40 atmosphere @ 16.8°C . (Possibly formed as a byproduct of MEA-agent neutralization reactions.)

4. Description of Operation.

a. On the morning of the first day of the test, 31 bottles, each containing 25 ml of agent absorbed on 45 gm of activated charcoal, will be emptied into a 15-gallon drum containing 6 gallons of MEA. (The distribution of the agent bottles is as described in section 2.) After the last bottle has been emptied into the drum, an additional 3.4 gallons of MEA will be added, and the drum will be closed. The empty bottles will be rinsed with a 5% HTH solution, placed in a plastic bag which will be tied shut, and placed inside a 30-gallon steel drum for transport to "O" Field for disposal.

b. Each morning of the test, beginning with the first day, the following sequence of operations will be followed:

(1) The drum will be placed on the electric-powered drum roller and rolled for 1 hour (between 0830 and 0930 hours); pressure and temperature will be checked every 20 minutes. See Safety Procedures - (9) of this appendix.

(2) After drum has been rolled for 1 hour, the roller will be stopped, and two samples (50 ml each -liquid only) will be taken from the MEA solution in the drum, through the ball valve. These samples will be held in two 60-ml glass bottles. The drum will be placed in an upright position during the hold period. NOTE — The ball valve will be opened slowly in case of gauge failure.

(3) These two samples will be sealed with PARAFILM, placed inside a 5-1/2 pint tin can filled with vermiculite, and this can will also be sealed with PARAFILM.

(4) At 1000 hours, the samples will be transferred by Technical Escort Center personnel to building E3330, room 299, for analysis of HS present.

(5) The drum will be allowed to sit upright for 5 hours (between 0930 and 1430) and then will be placed on the drum roller and rolled for 1 hour (1430 to 1530). The drum will be placed upright and remain stationary until the following workday morning.

(c) When analytical results indicate that the amount of HS present in the MEA solution is 5 ppm or less, the daily routine of tests will be stopped. The drum will be rolled for 1 hour to allow complete mixing prior to sampling. Three samples will be drawn according to the procedures described below:

(1) Only 1 gallon of the MEA solution will be collected in a glass container. This sample will be drawn as described in 4.b(2). The container will be sealed with PARAFILM, placed in a 5-gallon pail with vermiculite and the lid on the pail crimped shut and sealed. This sample will be transferred via Technical Escort Center to Biomedical Laboratory, Environmental Toxicology Branch, building E3266 for analysis.

(2) Another 1-gallon sample will be collected into a wide-mouthed jar. This sample will be drawn as described in 4.(b)(2). This sample will include charcoal which will then be placed into the ball mill located in Lab II. The charcoal will be ground in the mill until a particle size range of 2 to 5 microns is attained. (This size range will be determined by drawing off a small sample of the milled material, and transferring the sample via Technical Escort Center to building E3330, room 299, for microscopic analysis. More than one sample may have to be taken and analyzed.)

(3) The drum will then be placed in the upright position, the bung will be removed, and a core sample of the charcoal will be drawn off after setting, using the core sampler. This sample will be placed in a suitable glass bottle, the bottle will be sealed, placed in a tin can with vermiculite, which is also sealed, and transferred via Technical Escort Center to building E3330, room 299.

5. Hazards Involved in the Operation.

NOTE: In the event any personnel are accidentally exposed, call Toxic Exposure Aid Station (TEAS) on extension 2008 or 17. First-aid treatment will then be initiated and the spill will be decontaminated as follows:

a. HS, HN-1, and HN-3 Mustard and Nitrogen Mustards.

A blister agent highly irritant to eyes, skin, and lungs; the material causes severe burns. Material spilled in the area will be decontaminated immediately with HTH solution followed by

water wash. First aid treatment of operations personnel at the work site will be with M5 solution if it is available for all areas except for in and around the eyes. Exposure casualties will be transported to Toxic Exposure Aid Station for treatment by the medical officer.

b. L. Lewisite.

Produces effects similar to HS, HN-1, and HN-3 but also acts as a systemic poison and may cause pulmonary edema, diarrhea, restlessness, weakness, subnormal temperature, and low blood pressure. Skin burns are much deeper than with H. Area spills will be decontaminated immediately with 5% HTH solution followed by flushing with water. First aid treatment of exposure victims is the same as cited in 5a.

c. PS, Chloropicrin

Acts as a vomiting agent, a choking agent, and a tear agent. Area spills will be immediately decontaminated using MFA followed by flushing with water. First aid treatment is immediate flushing with copious amounts of water, removal from contaminated area, and transportation to TEAS for treatment by the medical officer.

d. Safety Considerations.

a. Safety Equipment.

(1) The operations will be conducted in Lab II which is located on the first floor of building E 5625. This laboratory is ventilated by a 5,000 ft²/min exhaust fan which draws the air through a charcoal filter (C23R1).

(2) An M12 filter canister will be fitted to the nipple extension of the ball valve on the 15-gallon drum. This ball valve will be open at all times when the drum is in the upright position serving as a vent.

(3) Lab II is equipped with a water shower, a water hose, a caustic hose, and a floor drain which empties into a toxic sump. A pan of 5% HTH solution will be provided for washing boots, gloves, etc., which may become contaminated. A 5-gallon container of MFA will also be available to decontaminate any PS spills.

(4) During operations all personnel will be dressed in level A protective clothing as: long underwear, M3 impermeable suit and boots, hooded M9 protective field mask, M4 butyl rubber gloves with impregnated glove liners, and impregnated socks.

(5) A differential pressure gauge will indicate satisfactory operation of Lab II ventilation system.

(6) A CO₂ fire extinguisher is on hand.

(7) All electrical motors and wiring in Lab II are class 1, division 2, Groups B and D, and are grounded.

(8) Telephone, intercom, and emergency siren facilities are provided.

(9) An ambulance can be summoned by calling extension 2008 or 17. This ambulance will transport any casualty to Toxic Exposure Aid Station for treatment after decontamination operations are completed.

b. Safety Procedures.

(1) All personnel involved in the test operations, the decontamination of equipment used, and anyone required to enter the "V" room of Lab II where these operations are being conducted will wear level A protective clothing as described in paragraph 6a(3) above. Authorization for entrance to this room will be granted only by the Chief, Pilot Plant Section or the engineer in charge of the test. This authorization will be granted only to those personnel previously cleared by the Civilian Employees' Health Clinic for working in level A clothing.

(2) Prior to entering into the operating room of Lab II, the engineer in charge will:

(a) Ensure that each man is properly clothed in level A protective clothing. Masks will be tested with amyl acetate (banana oil) to ensure proper fit, gloves, suits and boots will be tested for leaks, and inspected for tears, holes, etc.

(b) Have suitable level B protective clothing available to assist if emergency action is required.

(3) Before leaving the operating room of Lab II, the operators will shower with the decontamination shower and will wash their gloves with 5% HTH solution, water, EMA, and water, in that sequence. This is to prevent accidental contamination of any other area.

(4) Any contaminated clothing, equipment, or floors will be thoroughly washed with 5% HTH solution, flushed with water, washed with MEA, and flushed with water. The clothing which has been decontaminated in the above manner will be placed in a plastic bag which will be tied shut and then placed inside a sealed drum for later disposal by Technical Escort Center personnel.

(5) Should a fire occur, it will immediately be extinguished using the CO₂ fire extinguisher.

(6) Should the ventilation system fail, personnel in Lab II (non-toxic area) will don masks immediately. If ventilation cannot be restored and the engineer in charge determines that a hazardous condition exists, all personnel in the building will be alerted by siren, and the general emergency procedures (appendix A) put into effect.

(7) Toxic Exposure Aid Station personnel will be notified by telephone extension 2008 prior to commencing any operations with agent, and after these operations are completed.

(8) For first aid procedures, see appendix A.

(9) If pressure rises above 10 psi and/or the temperature rises above 150°F, the drum roller will be shut off and the drum will be placed upright. The valve will be opened slowly and the drum will be allowed to vent to the area filters. This will be done in full level A clothing.

APPENDIX C
HAZARD CLASSIFICATION OF MATERIALS

Chemical Laboratory

18 August 1976

DISPOSITION FORM

For use of this form, see AR 340-15; the proponent agency is The Adjutant General's Office.

REFERENCE OR OFFICE SYMBOL SAREA-CL-TE	SUBJECT Hazard Classification of Materials
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TO Mfg Tech Dir Attn: SAREA-MT-E/Mr. Rosenberg	FROM Dir of Cml Lab	DATE Mr. Owens/ab/2129	CMT 1 18 AUG 1976
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1. Reference is made to:

a. CSPCS Work Package-Item Nomenclature: Ultimate Disposal (ULT), Project/Task Number 6S-05-01/728012.21, Work Breakdown Structure Title: MEA Detox Study-Navy Sets, Work Package Control No. 6-5 dated 24 May 1976.

b. Expenditure Order Assignment/Program dated 10 June 1976, Tech Project 728012.21 PRON A16156501R01F4, Appropriation No. 2162020, Cost Center 332, Exp Order 6S-05-KT, Program Amount: \$4,000.

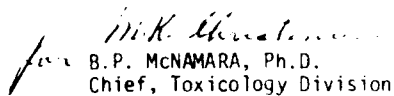
2. Toxicity studies as devised in CFR Title 49 (174.343) were performed to determine the hazard classification of the following materials:

a. H, L, PS decontaminated with MEA (Sample number DE50).

b. H, L, PS Ball Milled (Sample number DE51).

3. The results of the prescribed testing of the kit components showed none of the materials meet the Class B poison criteria as defined by CFR Title 49. The materials proved to be less toxic than the standards cited in the regulation, by all routes of administration. The hazard classification of CORROSIVE LIQUID would be more appropriate for each of the items.

FOR THE DIRECTOR:


B.P. McNAMARA, Ph.D.
Chief, Toxicology Division

DA FORM 2496
1 FEB 62

REPLACES DD FORM 98, EXISTING SUPPLIES OF WHICH WILL BE
ISSUED AND USED UNTIL 1 FEB 63 UNLESS SOONER EXHAUSTED.

☆ U.S. GPO: 1974-555-130/9000

APPENDIX D
RESIDUAL MUSTARD IN MONOETHANOLAMINE-TREATED CHARCOAL

Manufacturing Technology Directorate

1 December 1976

Purpose.

To further establish the efficiency of the monoethanolamine (MEA) system for demilling X-sets with agent on charcoal, another test was made. The objective was to determine the amount of residual agent left on charcoal after extensive MEA treatment.

Approach.

Add mustard to activated charcoal, react the impregnated charcoal with MEA, and then sample the mixture to determine the residual mustard with respect to time. The results would indicate the presence of mustard in parts per million over a period of time.

Procedure.

It was necessary to prepare a fresh laboratory sample of mustard (HD) and charcoal for the test because no "X" sets were available at Edgewood Arsenal. The sample consisted of 0.25 liters of HD added to 0.9 liters of activated charcoal (12-30 mesh) in a glass jar. The sample was capped and stirred to insure that the mustard had time to become adsorbed; it was kept at room temperature for more than 60 days. The mustard-impregnated charcoal was then added to a 22-liter drum containing 11.5 liters of MEA. The drum was stored at room temperature and rolled 1 hour per day, 5 days a week, for a 30-day period. The HD and MEA ratio was adjusted to the ratio planned for the demilitarization of X sets at Johnston Island. Fifty liter samples of MEA and charcoal were then removed periodically and analyzed for the presence of mustard. Sample analysis began upon receipt of the samples.

Three types of analyses were made:

1. The MEA solutions were diluted with aqueous NaCl and extracted with 1 part hexane to 10 parts of solution to concentrate the mustard by a factor of 10. The hexane was analyzed by gas chromatograph (GC) by comparison with standard solutions of HD in hexane.
2. The charcoal samples were analyzed by taking 30 to 35 grams of the MEA-saturated charcoal and washing it with 30 ml of chloroform (CHCl_3). The CHCl_3 was drained and analyzed by GC. Two more CHCl_3 washes were performed and analyzed in the same manner for mustard. The mustard reported from each wash was added to yield the CHCl_3 extraction analysis.
3. A portion of the CHCl_3 -extracted charcoal was then placed under vacuum, and any distillate was collected in a liquid nitrogen trap. The charcoal was then heated to 180°C , still

collecting the distillate in the trap. The distillate, approximately 1 to 2 ml of CHCl_3 , was removed from the vacuum line and analyzed by GC. The results of these analyses are tabulated below. A report of zero ppm indicates that the mustard concentration was less than the limits of detection. For the MEA solutions, the limit is 0.1 ppm, while the limit for the CHCl_3 solutions is 1 ppm.

Samples were analyzed on a Hewlett Packard Model 5830 recording gas chromatograph. The stainless steel column, 6 foot by 1/8 inch, was packed with 10% UCW982 on 80/100 mesh Chromosorb WHP. The temperatures were: Column - 160°C , injector - 250°C , and detector - 300°C . Helium was used as a carrier gas at a flow of 30 ml/min for the flame ionization detector.

Results.

The results of the analysis are shown in the following table:

Table. HD Recovered from Charcoal

Time of sample after start of test	HD in MEA solution	HD recovered from CHCl_3 extraction of charcoal	HD recovered from charcoal by vacuum distillation
days	ppm	ppm	ppm
5	0	—	—
8	0.3	—	—
9	—	3	0
15	0	—	—
18	—	0	0
19	0	—	—
23	—	0	0
31	0	2	3
67	—	0	0

Discussion.

The results show that mustard was present in the charcoal 10 days after the start of the test; but no mustard could be detected in the charcoal on the 18th and 23rd days. Then, on the

31st day, mustard was present in the charcoal. On the 67th day, the analysis was repeated, but no mustard was recorded in the charcoal. Based on the above and earlier tests, it can be concluded that mustard can be effectively removed from charcoal when using MEA as the decontaminating solution.

APPENDIX E
PROPOSED FULL-SCALE PROCEDURE

Manufacturing Technology Directorate
1 December 1976

Using the pilot study as the model for the "on-site" demilitarization system, a demilitarization procedure using scaled-up equipment was developed. The figure shows a detailed procedure for handling and detoxing the K958/"X" sets. The procedure utilizes a section of the Edgewood Arsenal chemical neutralization system for disassembling, storing, and demilling agent. The disassembly area is used for dumping the agent-impregnated charcoal from the set bottles into a standard 55-gallon drum. After the agent is added to monoethanolamine (MEA), the drum is removed and bunged with a filter assembly and with a temperature-pressure probe. The drum containing agent and the drum containing dunnage are then moved from the reactor site to be rolled daily (28 days maximum) to insure that the contents (basically charcoal) are thoroughly mixed and reoriented in the liquid detoxification solution.

The components designed specifically for handling the X-sets are as follows:

1. The filter is for the 55-gallon drum containing the agent and MEA. The filter is an M11 and it acts on an agent scrubber if the vapor pressure within the drum exceeds the sensitivity of the diaphragm valve. The diaphragm valve will contain the ambient (65° to 100°F) vapor pressure of MEA within the drum thereby preventing premature saturation of the filter. The globe valve, assembled to the stem, closes the stem to the filter when the drum is about to be placed horizontally on the drum roller.
2. The feed funnel, fabricated from polyethylene, is a commercial item having a 2-inch-diameter spout. The funnel assists the operator while he pours agent-impregnated charcoal into the 55-gallon drum.
3. Drum top seal, a 50-60 drumometer 1-inch-diameter rubber disc, is placed on top of the 55-gallon drum during agent transfer. The seal provides an airtight interface between reactor drum and glove box.
4. Temperature and pressure probe assembly is threaded to the large bung hole of the agent and MEA drum. The gauges read the condition of the MEA solution when the reactor drum is in a horizontal position on the drum roller.
5. Pliers are used for manually removing the wax/cheesecloth bottle seal and cap from the bottles containing agent-contaminated charcoal.

A list of the equipment necessary for the demilitarization of the X and K955 sets at F Field is as follows:

- a. Level D clothing and gas mask

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- b. Filtered glove box and set storage area
- c. Disassembly tools
- d. Drum fork lift
- e. Drum roller
- f. 55-Gallon drum (5 each)
- g. 140 Gallons of MEA
- h. Record sheet
- i. Sampler dipper
- j. Process timer
- k. Assorted wrenches
- l. Drum pump with metering valve
- m. Drum heater
- n. Balance scale 0 to 500 gm (min)
- o. Corrosive liquid and labels

It is required that MEA drum temperatures be maintained in the range of 65° to 100°F throughout the demilitarization process. Drum band heaters are utilized to prevent the MEA from solidifying when ambient temperatures drop below the freezing temperature of MEA (57°F).

If while rolling, the drum temperatures and/or pressures approach 125°F and 5 psig, respectively, stop rolling, stand the drum upright, and slowly open the filter valve. Replace the M11 filter after pressure and/or temperature have returned to ambient.

Weigh the M11 filter once every 2 days and replace it when its "as used" weight exceeds its original weight by 25 grams.

Maintain records of what is added to each drum. Drums are to be coded for cross reference with written records.

This procedure requires the proper field testing before it is implemented.

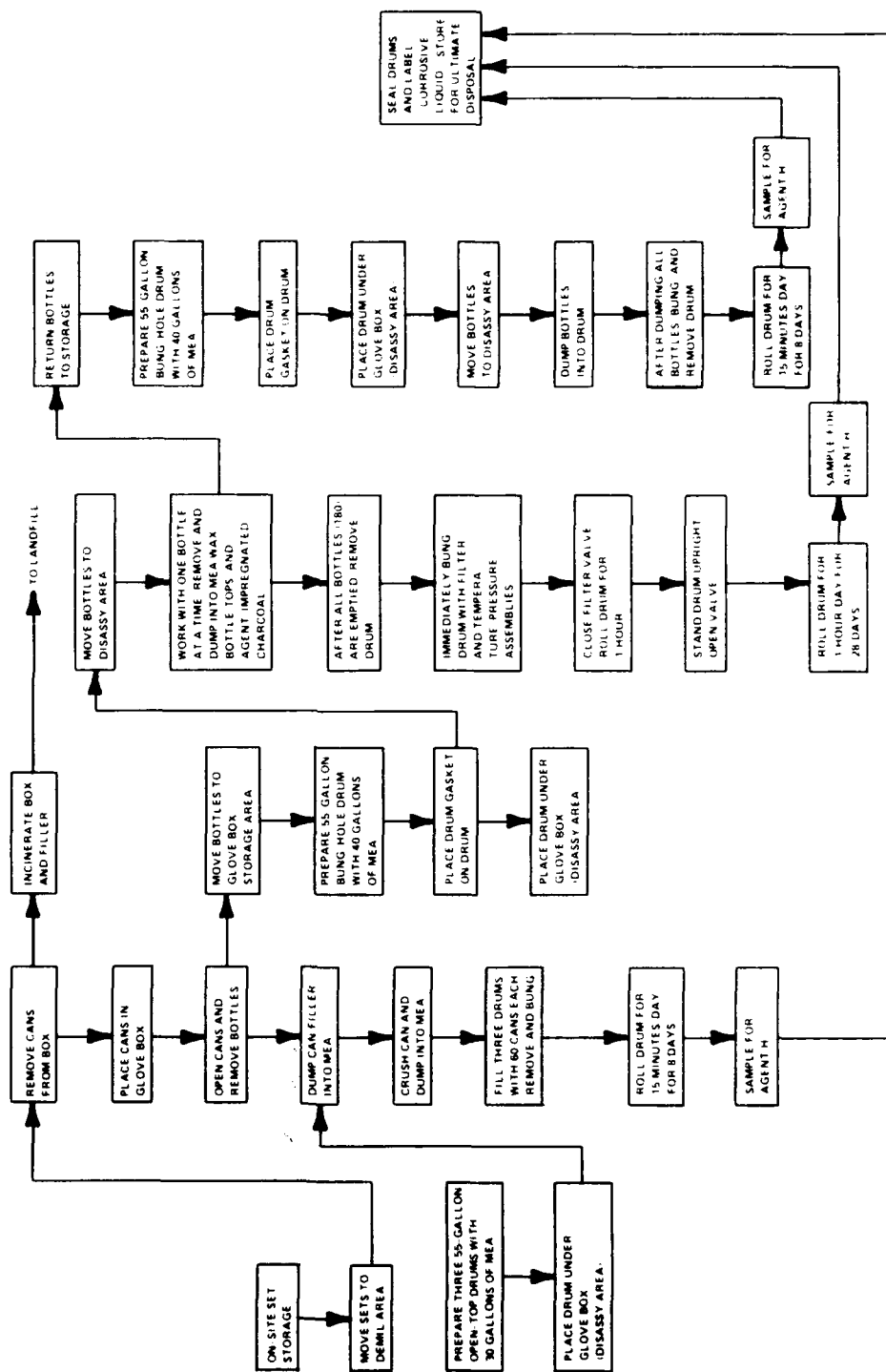


Figure. Process Plan for Detoxing K955 and X-Sets

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